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By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of:-

ISP INVESTMENTS INC.
Incorporated in USA - Delaware
300 Delaware Avenue
Suite 303
Wilmington
DE19801
United States of America

ADP No. 08390122001

Patents Form 1/77

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Request for grant of a patent

19 APR 2002

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1.	Your reference	7188 GB/JR/JSnr/Chick Feed
2.	Patent application number *(The Patent Office will fill in this part)*	0208999.3
3.	Full name, address and postcode of the or of each applicant *(underline all surnames)*	ISP Alginates (UK) Ltd. Waterfield Tadworth Surrey KT20 5HQ SECTION 30 (1977 ACT) APPLICATION FILED 14.05.02
	Patents ADP number *(if you know it)*	806326500 1
	If the applicant is a corporate body, give the country/state of its incorporation	England and Wales
4.	Title of the invention	Process, Equipment and Products
5.	Name of your agent *(if you have one)* "Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*	Abel & Imray 20 Red Lion Street London WC1R 4PQ
	Patents ADP number *(if you know it)*	174001

		Country	Priority application number *(if you know it)*	Date of filing *(day/month/year)*
6.	If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number			

		Number of earlier application	Date of filing *(day/month/year)*
7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application		

8.	Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:* *a) any applicant named in part 3 is not an inventor, or* *b) there is an inventor who is not named as an applicant, or* *c) any named applicant is a corporate body.*	Yes

9.	Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document.	
	Continuation sheets of this form	
	Description	11
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10.	If you are also filing any of the following, state how many against each item.	
	Priority documents	
	Translations of priority documents	
	Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
	Request for preliminary examination and search *(Patents Form 9/77)*	One
	Request for substantive examination *(Patents Form 10/77)*	
	Any other documents *(please specify)*	
11.		We request the grant of a patent on the basis of this application. Signature: Abel & Imray ABEL & IMRAY Date: 19 April 2002
12.	Name and daytime telephone number of person to contact in the United Kingdom	Janet Senior 020 7242 9984

ISP Alginates (UK) Ltd. 7188GB/Chick Feed

"Process, Equipment and Products"

ABEL & IMRAY
Chartered Patent Attorneys
20 Red Lion Street
London WC1R 4PQ

JR/JSnr/CL

Process, Equipment and Products

Alginate gels are well-known as are methods for their preparation by converting alginate as its sodium salt form, as a sol, into a gel by action of calcium ions.

Alginate gels have been used as foodstuffs e.g. petfoods with meat in alginate gel and simulated fruits with pureed fruit in alginate gel.

Alginate sols are usually in the form of the sodium salt but other cations can be used to form sols. Similarly calcium ions are usually the cations used to convert the sol to a gel form but other cations can be used. Suitable other cations for the sol and other cations/salts for conversion to gel form are described below. Similarly it is well-known that low-methoxy pectate has very similar characteristics to alginate as explained below but we will describe the background to our process and products initially only in terms of sodium alginate and use of calcium ions to gel the sol.

Broadly speaking there are three methods of converting an aqueous sol of sodium alginate to a gel. The first is by diffusion of calcium ions into an aqueous sol of sodium alginate. The second is by diffusion of hydrogen ions i.e. from an acid into an aqueous sol of sodium alginate containing a calcium salt the solubility of which is greatly increased by the hydrogen ions. The third is by mixing an aqueous sol of sodium alginate with a source of soluble calcium ions and allowing the mixture to gel without further mixing.

To obtain good quality gels it is important that gelation occurs as far as possible in the absence of shear.

The third method has the major disadvantage over the other two methods that the sodium alginate sol is mixed with a source of soluble calcium ions and shearing at least at the start of gelation is unavoidable. All the methods of preparing acceptable gels have involved use of relatively complex systems and have required skills above that of an untrained person. We have invented a process and related equipment by which the third method can be used to prepare acceptable gels and in particular gel pieces with especially useful charateristics, even without the use of complex systems.

In a particular form of our invention our process and related equipment and products can be used for delivering therapeutic amounts of biologically active substances to humans and animals and, more particularly, to livestock.

An aspect of our invention is that we have discovered that alginate and low-methoxy pectate sols can advantageously be produced by adding a dispersion of alginate or low-methoxy pectate to water in an in-line dynamic mixer. Therefore our invention, in this aspect, provides a process for preparing an alginate or low-methoxy pectate sol in which a dispersion of alginate or low-methoxy pectate is mixed with water in an in-line dynamic mixer.

In a further and particularly important aspect of our invention we provide a process in which water and a

dispersion of alginate or low-methoxy pectate are mixed in an in-line dynamic mixer to produce a sol of hydrated alginate or low-methoxy pectate and then free gelling ions are generated in the sol either a) by including in the water or in the dispersion of alginate or low-methoxy pectate a salt providing gelling ions when dissolved which salt is insoluble at neutral pH but soluble at acid pHs and by feeding an acid to the sol as an aqueous solution or as a dispersion or b) by feeding a dispersion of a salt providing gelling ions to the sol after which the resulting mixture is allowed to gel. Alternative b) is preferred. It is most unexpected that gelling ions can be generated and mixed with alginate or low-methoxy pectate sol in an in-line dynamic mixer without the resulting gel being of poor quality. It is also most advantageous in that it provides a simple process for preparing alginate or low-methoxy pectate gels.

The alginate or low-methoxy pectate is preferably dispersed in an anhydrous liquid dispersant which disperses or dissolves in water. The suspending liquid dispersant should preferably be such that alginate or low-methoxy pectate can remain in suspension in it over periods of up to fifteen minutes without stirring. The liquid dispersant should also preferably have lubricating properties e.g. to be pumpable in conventional progressive cavity pumps. Such lubricating properties are less important when piston pumps are used. Examples of suitable liquid dispersants are oils, glycerol and polyols. Advantageously the oil can be

an edible oil containing lecithin e.g. a vegetable oil containing about 10% lecithin. Use of edible oils is advantageous when the process is used to prepare a gel for feeding to livestock.

The low-solubility salt providing gelling ions, i.e. the one used in alternative b), is preferably dispersed in a liquid which disperses or dissolves in water. Its viscosity characteristics should preferably meet the criteria set out for the dispersant for the alginate or low-methoxy pectate. The dispersant for the salt providing gelling ions is preferably anhydrous and it should be noted that water itself would produce a slurry which could not be pumpable in conventional progressive cavity pumps.

A feature of our invention is that there is reduced need to use calcium sequestrants.

In a specific form our process provides an advantageous system for preparing alginate or low-methoxy pectate gels containing therapeutic amounts of biologically active substances, including but not limited to vitamins, enzymes and bacteria, especially those which are best kept in a protected environment e.g. dry or anaerobic till they are fed to patients or livestock.

In a special aspect of our invention such therapeutic amounts of biologically active substances can be incorporated in the dispersion of alginate or low-methoxy pectate or in the water or in the aqueous acid or dispersion of low-solubility salt providing gelling ions depending on the sensitivity of the active substance to

water and to acid.

It is also convenient to incorporate other components in the dispersion of alginate or low-methoxy pectate or the dispersion of salt providing gelling ions. For instance this obviates the need for using very small dosing pumps. It also helps prevent settling out of the salt providing gelling ions.

An advantage of our process is that it can be performed at ambient temperature, in particular at low ambient temperatures i.e. at temperatures below 30°C especially below 20°C. Of course the temperature must be above freezing point i.e. above 0°C. Use of low temperatures helps avoid deterioration of active ingredients e.g. heat-sensitive ingredients such as biologically active additives useful for optimal health and nutrition.

A further advantage of our process is that it can achieve the uniform incorporation of attractants which e.g. can substantially increase the probability of consumption by the livestock e.g. green colour for chicks and species-specific attractants in the fishing industry e.g. in fish farming. When being used to prepare gels for use in fish farming it is advantageous to include air when mixing the dispersion of alginate or low-methoxy pectate with the water and/or when mixing them with the calcium-ion generating system.

As mentioned above our process does not necessarily involve the use of complex systems or expensive equipment.

Thus a further advantage of our process is that our process and equipment can be operated on demand and by relatively untrained people on site using minimal equipment and, without e.g. requiring the preservation of ingredients in an active state during transport and storage.

Our invention can particularly advantageously be used to produce an alginate or low-methoxy pectate gel containing a sensitive ingredient which requires an aqueous environment and which requires to be fed, e.g. to livestock, shortly, e.g. within 30 minutes, after being introduced to an aqueous environment. Indeed we have found, although the products of our process are particularly advantageous in this respect, that alginate or low-methoxy pectate gels are excellent delivery media for such sensitive ingredients. Such gels deliver water, useful for the livestock and useful for certain biologically active materials e.g. bacteria but without excess free water, which can lead to problems e.g. hypothermia. Important examples of such sensitive ingredients are anaerobic bacteria and in a particularly important form of our invention a product is formed comprising anaerobic bacteria dispersed in alginate or low-methoxy pectate gel in which any water used is de-aerated water. For instance the water mixed with the dispersed alginate or low-methoxy pectate in our process contains dispersed anaerobic bacteria.

The water can be de-aerated by adding salts which generate carbon dioxide or simply by the addition of solid

carbon dioxide in which the anaerobic bacteria have been delivered. The former is preferred because salts can be used which contain minor ingredients which are beneficial to the anaerobic bacteria.

The ratio of alginate or low-methoxy pectate to calcium can be adjusted to get adequate dryness with lack of significant syneresis and adequate strength. The process enables attainment of fast setting times e.g. within about 11 minutes of addition or production of the gelling ions. Increasing the amount of the alginate or low-methoxy pectate and increasing the level of salt providing gelling ions increases gel strength and dryness. Increasing the amount of salt providing gelling ions, without increasing the amount of alginate or low-methoxy pectate, will speed up the gelling rate and increase the gel strength but speeding up the gelling rate too much will increase syneresis.

Preferred sizes are particles of about 1 to 4 mm in maximum dimension but larger particles can be used if they are sufficiently friable.

Equipment according to the invention consists of feed means through which a) a dispersion of alginate or low-methoxy pectate, b) water and c) a dispersion of a low-solubility salt providing gelling ions can be separately fed to an in-line dynamic mixer, the point of entry to the dynamic mixer of the feed means for a) and for b) being sufficiently spaced up-stream of the point of entry of the feed means for c) that in use the alginate forms a sol with

the water before it comes into contact with calcium ions.

As mentioned above for convenience we describe our process and products initially in terms of the sodium form of alginate as a sol and gelation using calcium ions from salt providing gelling ions but other cations can be used. Similarly we mention above that it is well-known that low-methoxy pectate has very similar characteristics to alginate.

"Low-methoxy pectate" is a well-known term. Normally low-methoxy pectates are considered to be pectates (i.e. pectins) containing less than 50% methoxylated hydroxyl groups. For the process of this invention the low-methoxy pectate should preferably contain less than 30% methoxylated hydroxyl groups.

As mentioned the sodium salt is a particularly convenient form of alginate or low-methoxy pectate from which to form a sol. The alginate or low-methoxy pectate used to form the products of the invention is preferably sodium alginate of high molecular weight (of the order of 100,000). Alginates having a low content of mannuronic acid residues (mannuronic: guluronic ratio less than 1:1) are especially suitable. The proportion of alginate or low-methoxy pectate used varies with its gelling ability (that is, the gel strength obtained per unit weight) and with the texture desired in the final product, in particular in the gel pieces. We have found that when the preferred sodium alginate is used it suitably forms from 0.4% to 4% by weight of the product formed. Other cations can be used to

form sols with alginate or low-methoxy pectate e.g. potassium and ammonium.

As mentioned, calcium sulphate (particularly in the dihydrate form) is the preferred low-solubility calcium salt to be used in the invention. However any calcium salt which has low solubility in water e.g. in the aqueous sol can be used. Salts with a solubility less than 3.5% (weight percentages) are preferred, particularly preferably those with a solubility less than 1% and especially those with a solubility less than 0.3% but above a solubility of 0.02% e.g. calcium sulphate anhydrous, calcium sulphate dihydrate, calcium citrate and calcium tartrate.

When the salt is a salt insoluble at neutral pH but soluble at acid pHs, preferred calcium salts include calcium citrate, calcium tartrate, calcium carbonate and calcium phosphates. Dicalcium phosphate dihydrate and dicalcium phosphate anhydrous are particularly preferred, especially dicalcium phosphate dihydrate.

Our process, equipment and products will be now described by way of example with reference to Figures 1 and 2.

Figure 1 is an overall flow diagram.

Figure 2 shows more detail of the gelling and dicing.

The quantities of ingredients were:

	% (by weight)
Water feed:	
De-aerated water	90
Anaerobic bacteria plus minor ingredients	0.56
Alginate feed:	
Sodium alginate (Manugel DMB*)	3.50
Oil blend**	3.00
Calcium sulphate feed:	
Calcium sulphate dihydrate	0.80
Chick feed	1.00
Oil blend	1.14
Total	100

* Trade Mark of ISP Alginates

** Oil blend: Vegetable oil 90%, lecithin 10%

Sodium alginate is dispersed in vegetable oil containing 10% lecithin. Anaerobic bacteria, supplied as beadlets packed in solid carbon dioxide, are dispersed in deaerated water. The beadlets contain micro-ingredients to help activate and increase the growth rate of the anaerobic bacteria. The water was deaerated by adding salts which generated carbon dioxide.

The dispersion of sodium alginate in vegetable oil containing 10% lecithin and the dispersion of anaerobic -

bacteria in water were fed to an in-line dynamic mixer M1 by pumps P1 and P2 at rates 108.67 kg/hr and 7.8 kg/hr respectively. The mixer M1 was a 2kw mixer and was operated at 2800 rpm. The mixer has a rotating shaft with four equally spaced rows of 8 pins, each with a tip to tip diameter of 66mm. The central shaft has a diameter of 35mm. There is one row of stators attached to the shell. The water and the dispersion of sodium alginate were fed to the in-line dynamic mixer through inlets aligned with the first rotating pin.

Calcium sulphate was dispersed in oil together with milled chick feed and fed by pump P3 at 3.53 kg/hr to the in-line dynamic mixer M1 beyond through an inlet aligned with pin 6. The alginate was hydrated i.e. in sol form by the time it reached pin 6 i.e. before being mixed with the calcium sulphate. The chick feed optimally contains a green colorant as this adds to the palatability of the product to chicks. The resulting mix was fed to moulds MD1 set in a carousel C1 which rotated at 11 minutes per revolution. At stage 10 the mix which by that time had gelled was ejected by an ejector E1 into a Hobart mixer, dicer D1, where it was broken down into particles of 3 mm diameter. The product was attractive and beneficial to chicks.

CLAIMS

1. A process for preparing an alginate or low-methoxy pectate sol in which a dispersion of alginate or low-methoxy pectate is mixed with water in an in-line dynamic mixer.

2. A process for preparing an alginate or low-methoxy pectate gel in which a sol of hydrated alginate or low-methoxy pectate is prepared according to claim 1 and then free gelling ions are generated either a) by including in the water or in the dispersion of alginate or low-methoxy pectate a salt providing gelling ions when dissolved which is insoluble at neutral pH but soluble at acid pHs and by feeding an acid to the sol as an aqueous solution or as a dispersion or b) by feeding a dispersion of a low-solubility salt providing gelling ions to the sol after which the resulting mixture is allowed to gel.

3. A process according to claim 2 in which the free gelling ions are generated by feeding a dispersion of a low-solubility salt providing gelling ions to the sol.

4. A process according to any one of claims 1 to 3 in which the alginate or low-methoxy pectate is dispersed in an anhydrous liquid which disperses or dissolves in water.

5. A process according to claim 4 in which the anhydrous

liquid is such that the alginate or low-methoxy pectate can remain in suspension in the liquid over periods of up to four hours without stirring.

6. A process according to claim 4 or claim 5 in which the anhydrous liquid has lubricating properties.

7. A process according to claim 3 and any one of claims 4 to 6 when dependent on claim 3 in which the low-solubility salt providing gelling ions is dispersed in a liquid that is capable of suspending the low-solubility salt providing calcium ions over periods of up to fifteen minutes without stirring.

8. A process according to claim 3 and any one of claims 4 to 7 when dependent on claim 3 in which the low-solubility salt providing gelling ions is dispersed in a liquid which has lubricating properties.

9. A process according to claim 3 and any one of claims 4 to 8 when dependent on claim 3 in which the low-solubility salt providing gelling ions is dispersed in an anhydrous liquid.

10. A process according to claim 3 and any one of claims 4 to 9 when dependent on claim 3 in which the low-solubility salt providing gelling ions is a calcium salt.

11. A process according to claim 10 in which the calcium salt is calcium sulphate.

12. A process according to any one of claims 2 to 11 in which either one or both of the liquids used to disperse, respectively, the alginate or low-methoxy pectate or the low-solubility salt providing gelling ions is an edible oil.

13. A process according to claim 12 in which the edible oil is a vegetable oil.

14. A process according to claim 12 in which the edible oil contains 10% of lecithin.

15. A process for preparing an alginate or low-methoxy pectate gel according to any one of claims 2 to 14 in which therapeutic amounts of biologically active substances are included in one or more of the liquids mixed in the dynamic mixer.

16. A process according to claim 15 in which anaerobic bacteria are the biologically active substances and they are introduced into the mixer by incorporation into the water.

17. A process according to any one of claims 2 to 16 in which the gel is broken into portions.

18. A process according to claim 17 in which the portions are fed to humans or livestock.

19. A process according to claim 18 in which the time between the incipiently gelling mixture leaves the mixer and the portions of gel are fed to livestock is less than 15 minutes.

20. A process according to claim 18 or claim 19 in which the livestock are chicks.

21. A product of a process according to any one of claims 1 to 20.

22. A feedstock for livestock which require water but are sensitive to free water in their environment consisting of pieces of gelled material according to any one of claims 2 to 21.

23. A feedstock for chicks according to claim 22.

24. An in-line dynamic mixer with a feed point for a dispersion of alginate or low-methoxy pectate and a feed point for water both feed points being at one end of the mixer and a further feed point for a dispersion of a low-solubility salt providing gelling ions spaced downstream of the other feed points such that when the dispersion of

alginate or low-methoxy pectate and the water are fed to the other two feed points and the dynamic mixer is operated a hydrated sol of alginate or low-methoxy pectate is formed before the mixture comes in contact with the salt providing gelling ions.

25. Gelled material containing therapeutic amounts of biologically active materials in which the gelled material is alginate or low-methoxy pectate.

26. Gelled material according to claim 25 containing anaerobic bacteria.

27. A feedstock for livestock which require water but are sensitive to free water in their environment consisting of pieces of gelled material according to claim 25 or 26.

28. A feedstock for chicks according to claim 27.

29. Any new feature described herein or any new combination of herein described features.

30. A process or product substantially described herein with reference to the Example.

Fig.1.

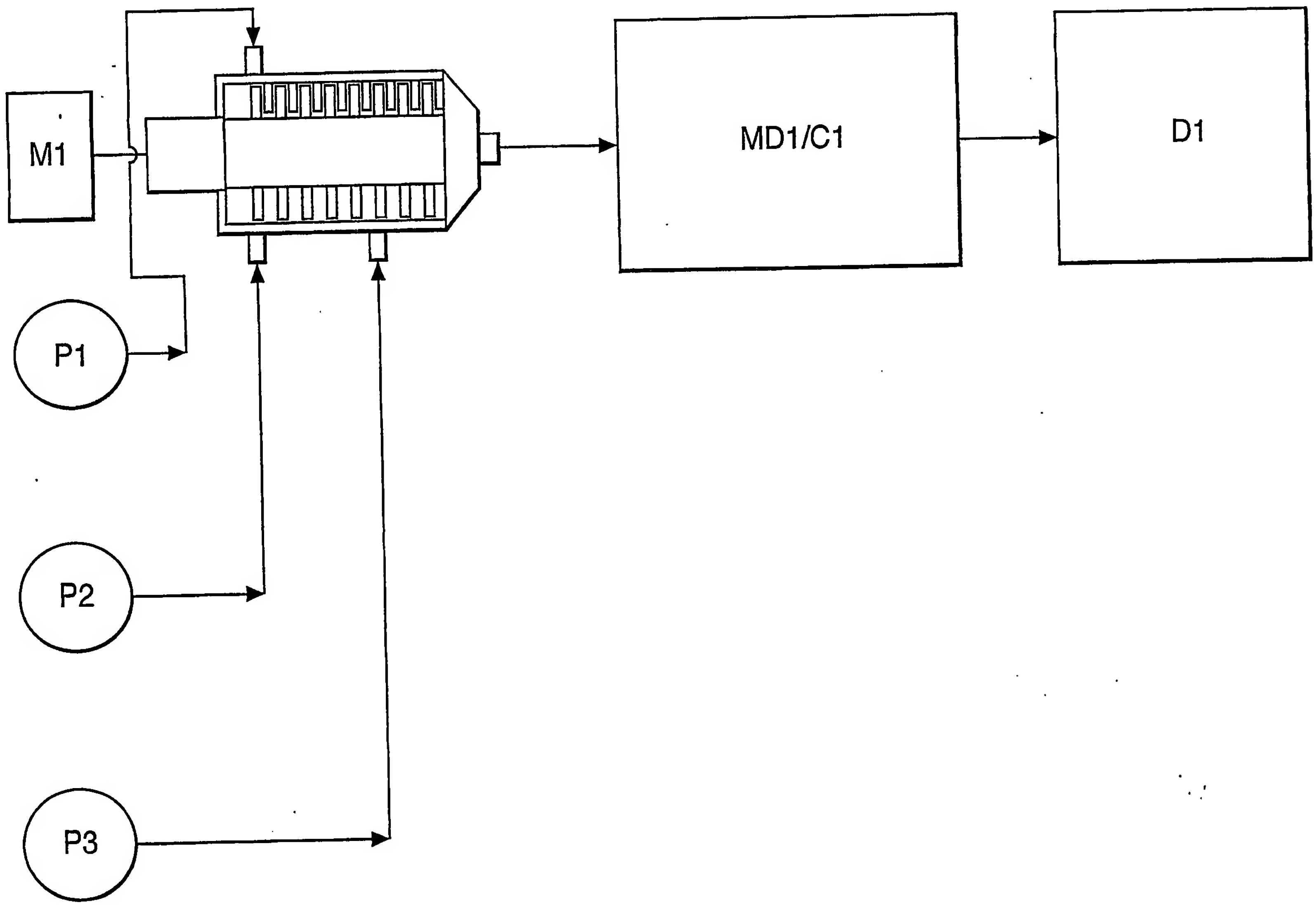
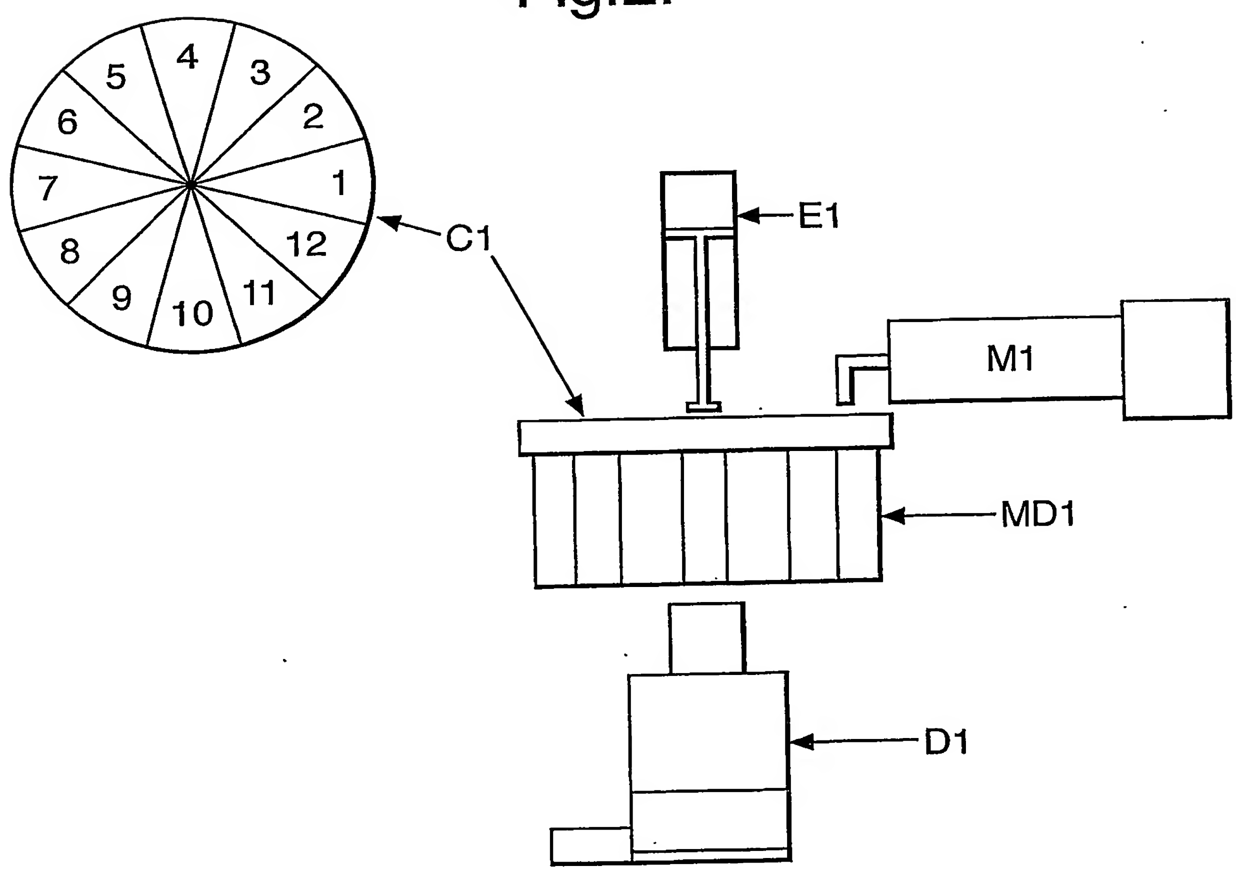


Fig.2.



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